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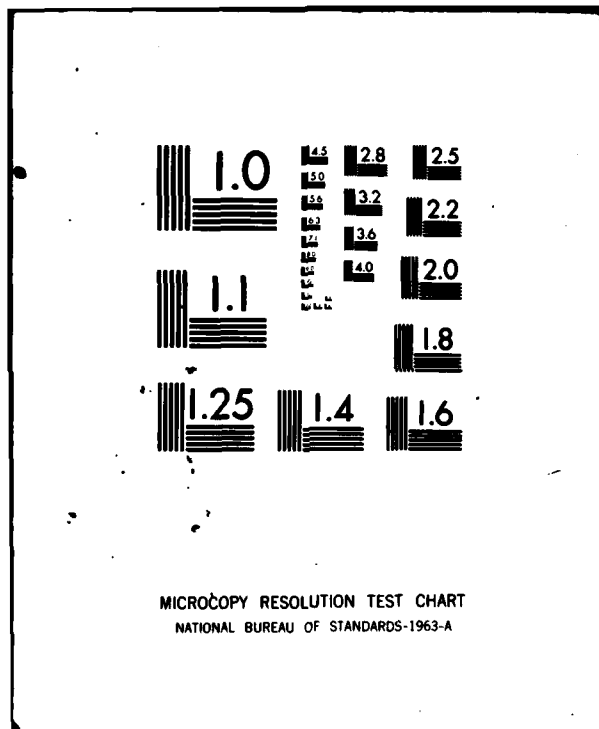
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**DETERMINATION OF ULTRAMICRO
QUANTITIES OF ELEMENTAL
PHOSPHORUS IN WATER BY NEUTRON
ACTIVATION ANALYSIS**

BY MING G. LAI
DAVID H. ROSENBLATT

RESEARCH AND TECHNOLOGY DEPARTMENT

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| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A highly sensitive method for the analysis of P ₄ in synthetic phosphy water has been developed. The method, using a neutron activation technique, has a detection limit of 0.01 PPB for P ₄ . In the procedure, elemental phosphorus is first separated from water by extraction into a suitable organic solvent such as benzene. P ₄ is then oxidized to phosphate, which is back-extracted | | |

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20. Abstract (continued)

into the aqueous layer. The amount of phosphorus is determined by thermal neutron activation analysis. A radiochemical separation procedure is used for the separation of phosphorus-32 activity induced during the neutron activation.

The method was tested on a series of synthetic phosphy water samples. P_4 recoveries, after corrected for blank, were found to be within 10% of the theoretical values for samples containing greater than 0.01 PPB P_4 . Less accuracy was obtained for samples of lower P_4 concentrations.

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SUMMARY

DETERMINATION OF ULTRAMICRO QUANTITIES OF ELEMENTAL
PHOSPHORUS IN WATER BY NEUTRON ACTIVATION ANALYSIS

In order to accurately assess the environmental consequences from the generation of "phossy water" wastes in loading operations at the Army Pine Bluff Arsenal, Arkansas, there currently exists an urgent requirement for a rapid and economical method for the determination of white phosphorus in water. This report describes the use of neutron activation technique for the determination of extremely low concentrations of white phosphorus in water. The method appears sufficiently sensitive for the characterization and monitoring of elemental phosphorus to meet the recommended discharge standard. The work was funded by the Army Medical Research and Development Command under Military Interdepartmental Purchase Request 5954.

*P. R. Wessel*P. R. WESSEL
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Chapter 1

INTRODUCTION

BACKGROUND

In order to accurately assess the environmental consequences from the generation of "phossy water" wastes in loading operations at the Army Pine Bluff Arsenal, Arkansas, there currently exists an urgent requirement for a rapid and economical method for the determination of white phosphorus (P_4) in water. Based on the extremely high toxicity of P_4 to the aquatic organisms, a discharge standard of less than 0.01 PPB has been recommended. Therefore, an analytical method, sensitive at least to that level, is required.

Until recently the usual method for determining P_4 involved extraction of the sample with an organic solvent such as benzene, oxidation of the extracted phosphorus to orthophosphate, which was then determined by the molybdenum blue or other colorimetric methods.^{1,2} Under ideal conditions, a detection limit of about 0.01 PPM of P_4 in water was attainable by this technique.

Recently, a more sensitive method has been developed for P_4 , employing a gas-liquid chromatographic technique.^{3,4} The method affords a minimum detectable concentration of about 0.2 PPB. Although the method is specific for P_4 , some interference from organics has been noted.

This paper describes a method that has been developed for the quantitative determination of P_4 in water using neutron activation analysis. The method is effective even when only 0.001 PPB of P_4 is present. In addition, the procedure is simple, rapid, and economical for routine analysis of large numbers of field samples.

1. Reiman, W. and Beukenkamp, J., "Treatise in Analytical Chemistry," Part 2, Vol. 5, Sect. A, Interscience, New York, N.Y., 1961.
2. Freimuth, H. C., "Toxicology," Vol. 2, Academic Press, New York, N.Y., 1961.
3. Ackman, R. G., and Addison, R. F., "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," Proc. Conf. Pollut., St. Mary's Univ., Halifax, N.S., Aug. 24-26, 1969.
4. Addison, R. F., and Ackman, R. G., "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," J. Chromatog, 47, 421 (1970).

NEUTRON ACTIVATION TECHNIQUE

The proposed method is based on the neutron activation technique. The technique can be simply stated. When any material is irradiated in a reactor, the activity induced in any given element can be calculated from the expression:

$$A = N\sigma\phi(1-e^{-\lambda t})$$

where

A = induced activity, dis/sec

N = number of target atoms present

σ = cross section, cm^2

ϕ = neutron flux, neutron/ cm^2 /sec

t = irradiation time

λ = decay constant of product nuclide

The reliability of the parameters in this relation is generally well-known with the exception of the neutron flux, ϕ . This is uncertain, and without detailed knowledge of the operation of the reactor throughout the period of irradiation there is no assurance of its magnitude or constancy.

The usual way to circumvent this difficulty is to irradiate along with the unknown, a small quantity of a pure compound of the element to serve as a standard. By comparison of the activities in the unknown and the standard, the amount of material in the unknown can be calculated.

Phosphorus lends itself particularly well to determination by neutron activation analysis. It has a fair activation cross section of 0.23 barn. Its half-life of 14.3 days is long enough to permit the decay of many possible interfering activities, but short enough to permit the ascertainment of radiochemical purity by half-life measurement. Moreover, it is a pure beta emitter and can be readily distinguished by absorption measurements or pulse-height analysis from most contaminants that have associated gamma emissions. If necessary, ^{32}P can be separated from other interfering activities by conventional radiochemical procedures.

APPROACH

The general approach of the method is shown in a schematic diagram (Fig. 1). Elemental phosphorus is first separated from water by extraction into a suitable organic solvent such as benzene. The P_4 is then oxidized to PO_4^{3-} , which is back-extracted into nitric acid. The amount of phosphorus is determined by thermal neutron activation analysis. A radiochemical separation procedure involving precipitation methods is used for the separation of phosphorus-32

activity induced during the neutron activation.

In order to expedite the experimental work for the development of the method, radioactive phosphorus tracers were used in the preliminary studies. These include:

1. Extraction efficiency of P_4 at different aqueous-benzene ratios
2. Stability of P_4 in benzene
3. Loss of P_4 during the evaporation process
4. Oxidation of P_4 to orthophosphate with nitric acid.
5. Back-extraction of oxidized phosphorus
6. Possible interferences from various sources

At very low levels of P_4 , contamination became a great problem. Therefore, large efforts were made to guard against all possible contaminations. This included purification of chemical reagents, removal of traces of phosphorus from experimental apparatus and containers and preparation of controlled blanks.

Chapter 2

EXPERIMENTAL

MATERIALS AND REAGENTS

1. White phosphorus with a purity of 99.999% was supplied by Ventron Corporation, Danvers, Massachusetts. The material was packed in argon atmosphere inside a sealed pyrex ampule.
2. Ultrapure nitric acid, supplied by Ventron Corporation had a certified phosphate impurity of less than 0.01 PPM. Actual analysis showed that concentration of phosphate was of the order of 0.001 PPM.
3. High purity benzene was obtained from Burdick and Jackson Laboratories, Inc. No detectable amount (< 0.01 PPB) of phosphorus was found upon analysis of the solvent.
4. High quality distilled water was obtained from National Bureau of Standards. The distilled water contained less than 0.001 PPB of phosphorus.
5. Standard carrier solution was prepared by dissolving $(\text{NH}_4)_2\text{HPO}_4$ in water to contain 10 mg of phosphorus per ml.
6. Molybdic acid reagent was prepared by dissolving 25 g of ammonium molybdate $(\text{NH}_4)_2\text{MoO}_4$ in 1 liter of water.
7. Magnesia mixture was prepared by dissolving 50 g of MgCl_2 and 100 g of NH_4Cl in 1 liter of water.

PREPARATION OF PHOSPHORUS STANDARD

Standard solutions of phosphorus were prepared by weighing white phosphorus in a glove box under inert atmosphere and suspending it ultrasonically in river water. Fresh standard solutions were prepared immediately prior to the start of each experiment.

EXTRACTION OF PHOSPHORUS

All extractions were carried out with high purity benzene. The mixture was equilibrated on a mechanical shaker or a magnetic mixer. The organic layer, containing P_4 , was separated in a separatory funnel and washed once with an equal volume of distilled water.

BACK EXTRACTION OF PHOSPHORUS

Treatment of the benzene extract by shaking with aqueous nitric acid resulted in nitric acid oxidation of P_4 to phosphate ion, which then passed into the aqueous phase. The treatment was carried out in a mechanical shaker or magnetic stirrer. The aqueous layer, containing phosphate, was isolated in a separatory funnel.

SAMPLE PREPARATION FOR IRRADIATION

The nitric acid solution obtained from the back-extraction was gently evaporated to about 0.2 ml on a hot plate under filtered air or nitrogen in a clean glove box. The solution was then transferred into a polyethylene vial (8mm diameter x 13mm length). The vial was sealed by welding with a hot glass rod. The heat-sealed container was then placed in a plastic envelope which was heat-sealed with a hot iron. Ten samples were placed in an NBS capsule and irradiated at the NBS reactor at a flux of $5.6 \times 10^{13} \text{ n/cm}^2\text{-sec}$ for a period of one hour.

RADIOCHEMICAL SEPARATION OF PHOSPHORUS-32

After irradiation the container was opened, and the liquid was transferred to a 50 ml centrifuge tube, diluted to 10 ml with 1M HNO_3 , and 1 ml of phosphorus carrier added. Then 15 to 20 ml of molybdic acid reagent were added and the solution heated with stirring until the yellow ammonium phosphomolybdate was precipitated. The precipitate was centrifuged, washed with 1M HNO_3 , and dissolved in 1M NH_4OH . The solution was diluted to 10 ml in distilled water, made slightly acidic with HNO_3 , and cooled. The phosphorus was then precipitated as $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ by the addition of 15 ml of magnesia mixture and excess NH_4OH . The precipitate was centrifuged, washed with dilute NH_4OH followed by 95% alcohol, and filtered on a tared filtered disk of Whatman's No. 42 filter paper. It was dried in a drying oven for ten minutes at 110°C , weighed, and mounted for measurement of the beta radioactivity from ^{32}P using a low-background proportional counter. The radioactivity measured was corrected for decay and chemical yield.

Chapter 3

RESULTS AND DISCUSSION

EXTRACTION EFFICIENCY

It has been demonstrated that elemental phosphorus in aqueous suspensions deteriorates rapidly. Hence, the exact recovery of known weights of added phosphorus is impossible in any experimental system containing water. Studies of extraction efficiency are therefore performed by the method of comparison. Extractions of P_4 were carried out with different water-benzene ratios. The results were then compared to that of a standard considered quantitative in the extraction process. In addition, second extractions of the same samples were performed. The recoveries of successive extractions were compared.

The results of the extractions of P_4 with benzene are presented in Table 1. Between 95 - 98% of phosphorus is extracted in one step when the water-benzene ratio is ten or less. A second extraction recovers only a small proportion of phosphorus from an aqueous suspension. For practical purposes, therefore, one extraction should be adequate.

Since other phosphorus species, if extracted into benzene, would interfere with the analysis, it is important to examine the extractability of several phosphorus compounds that have been found among the transformation products of P_4 in water. The results (Table 2) show that extremely small quantities of the phosphorus compounds under study are extracted.

STABILITY OF P_4 IN BENZENE EXTRACT

The stability of the organic extract with respect to phosphorus was examined for a period of 42 days. The results show that the phosphorus concentration is essentially unchanged during storage in a closed container at ambient temperature.

Another experiment was made to study the effect of solvent evaporation on the stability of phosphorus in benzene. Fig. 2 shows that loss of phosphorus occurs during the evaporation process under argon atmosphere at room temperature. The loss becomes more severe when the organic extract is reduced almost to dryness. From this study it is concluded that P_4 cannot be concentrated in the benzene extract by evaporation.

5 Lai, M. G., and Rosenblatt, D. H., "Identification of Transformation Products of White Phosphorus in Water," Naval Surface Weapons Center, NSWC/WOL/TR 76-103, 31 Jan 1977.

OXIDATION AND BACK-EXTRACTION OF P_4

Irradiation of materials in the reactor requires that the sample must be either a solid or a stable, non-volatile solution of relatively small volume. Since evaporation of the benzene extract would result in a complete loss of P_4 , the strategy was to oxidize P_4 to phosphate, back-extract the phosphate into the aqueous solution,⁴ and then evaporate the solution into a small volume.

In the experiment, equal volumes of the benzene extract and nitric acid solution of varying concentrations were equilibrated for varying periods of time. Recoveries of the P_4 (in the form of PO_4^{3-}) in the aqueous solution are shown in Table 3.⁴ It is obvious that⁴ recovery of phosphorus is a function of HNO_3 concentration and equilibration time. Quantitative recovery was obtained when the benzene extract was equilibrated with nitric acid greater than 5.3M for one hour.

In order to minimize the possible contamination of the phosphorus impurity in nitric acid and to maximize the concentration factor, attempts were made to reduce the amount of nitric acid in the back-extraction. Higher benzene-to-nitric acid ratios were tested for the back-extraction of P_4 . The results (Table 4) show that over 90% of P_4 (in the form of PO_4^{3-}) is recovered in the aqueous phase with a benzene-to-nitric acid (5.3M) ratio of 10.

Reduction of the volume of the aqueous solutions was accomplished by evaporation in a closed system under filtered air or nitrogen. No significant loss of phosphorus was detected when the volume was reduced from 10 ml to less than 0.2 ml.

Chapter 4

EVALUATION OF THE DEVELOPED METHOD

On the basis of the above results, a method for the determination of ultramicro quantities of white phosphorus was developed. The method was tested on the analysis of a series of water samples containing 0.001 to 1.0 PPB of P_4 . These samples were made of filtered river water (pH 7.5) containing known amounts of suspended P_4 . All samples were simultaneously processed to avoid the correction of P_4 decay.

PROCEDURE

To one liter of the sample were added 100 ml of benzene, and the mixture was vigorously agitated for 30 minutes. The benzene layer was separated and washed with two 50-ml portions of distilled water. To the benzene extract were added 10 ml of 5M HNO_3 , and the mixture was agitated for two hours. The aqueous layer was separated and evaporated to almost dryness at low heat on a hot plate.

The solution was transferred to a polyethylene vial and then irradiated in the NBS reactor for one hour at a neutron flux of 5×10^{13} n/cm²-Sec. After irradiation, the radioactive phosphorus in the sample was separated and purified by the radiochemical procedure previously described.

RESULTS

A quantitative evaluation of the analysis of seven samples is presented in Table 5. P_4 recoveries, after correction for blank, were found to be within 10% of the theoretical values for samples containing greater than 0.01 PPB P_4 . Less accuracy was obtained for samples of lower P_4 concentrations. A blank analysis shows that the quantity of phosphorus introduced through reagents and contamination is of the order of 0.003 μ g of phosphorus. This relatively large blank correction would probably contribute a considerable error to samples containing less than 0.01 PPB P_4 .

Under the irradiation conditions used in this work, the neutron activation technique can detect phosphorus down to 0.001 μ g. However, the actual limit of detection of P_4 in water by using the entire procedure and a sample volume of one liter is set by the background ^{32}P radioactivity obtained due to contamination from the materials used and the nature of the samples. This blank phosphorus radioactivity, equivalent to 0.003 μ g in this case, probably cannot be reduced for the purpose of routine analysis because of economic constraints. Thus, taking this into consideration, we would set the practical lower limit of detection of P_4 in water by this method at 0.01 PPB. However, it is possible to extend the detection limit

to 0.001 PPB if necessary.

In conclusion, we have developed a rapid and highly sensitive method for the determination of P_4 by neutron activation. P_4 levels can be measured, in the presence of other inorganic phosphorus compounds, down to 0.01 PPB with an accuracy of about 10%. However, there exists an uncertainty of possible interferences caused by the presence of organic phosphorus in the water samples. Therefore, further studies should be implemented to determine the effect of organic phosphorus on the analysis.

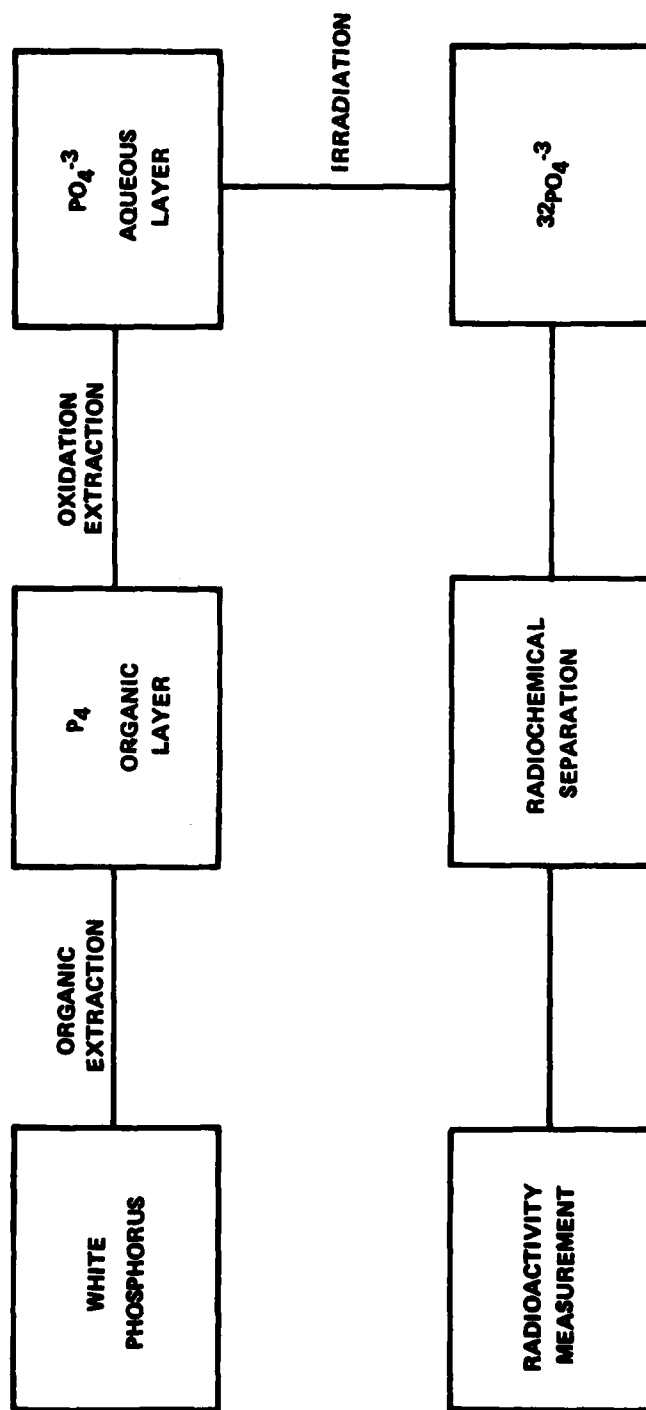


FIG. 1 FLOW DIAGRAM FOR THE GENERAL SCHEME OF THE NEUTRON ACTIVATION OF ELEMENTAL PHOSPHORUS IN WATER

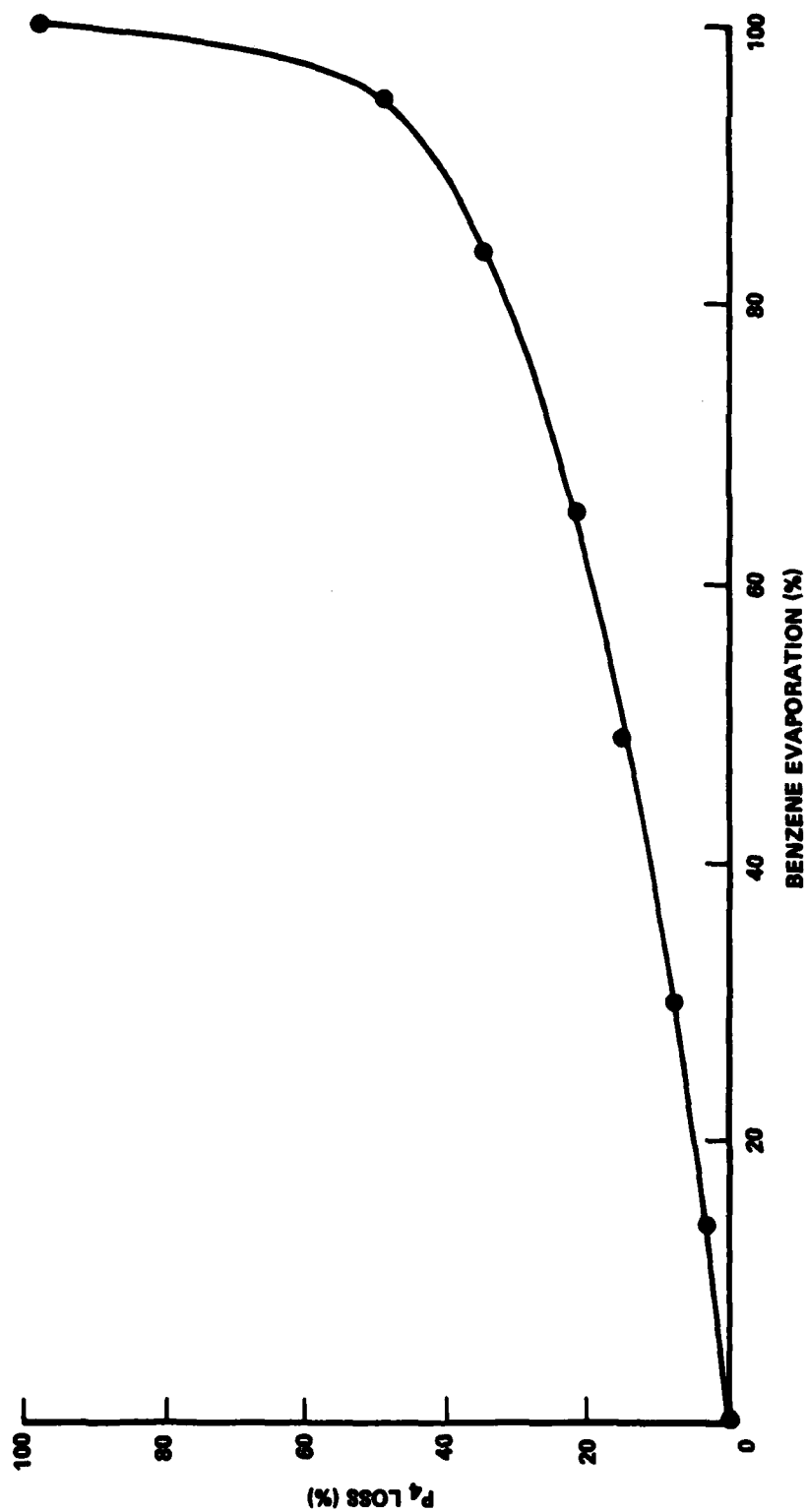


FIG. 2 STABILITY OF WHITE PHOSPHORUS IN BENZENE EXTRACT

TABLE 1

RECOVERY OF ELEMENTAL PHOSPHORUS
IN WATER BY BENZENE EXTRACTIONS

| WATER:: BENZENE (VOLUME) | PHOSPHORUS RECOVERED (%) | |
|-----------------------------|--------------------------|------------|
| | EXT. No. 1 | EXT. No. 2 |
| 1 * | 100.0 | 0 |
| 2 | 98.0 | 2.0 |
| 5 | 96.2 | 3.1 |
| 10 | 95.1 | 4.2 |
| 20 | 67.4 | 22.1 |

* COMPARATIVE STANDARD

EQUILIBRATION TIME : 30 MIN

TABLE 2

EXTRACTABILITY OF PHOSPHORUS COMPOUNDS
WITH BENZENE

| COMPOUND | AMOUNT EXTRACTED (%) |
|---------------|----------------------|
| HYPOPHOSPHITE | 8.0×10^{-4} |
| PHOSPHITE | 1.8×10^{-3} |
| PHOSPHATE | 8.0×10^{-4} |

SODIUM SALTS ; 10 mg/l

TABLE 3

RECOVERY OF PHOSPHORUS IN
NITRIC ACID SOLUTIONS

| NITRIC ACID (M) | PHOSPHORUS RECOVERED (%) | | |
|-----------------|--------------------------|--------|--------|
| | 5 MIN | 30 MIN | 60 MIN |
| 1.6 | 11.6 | 15.9 | 21.1 |
| 3.2 | 14.0 | 42.5 | 62.8 |
| 5.3 | 30.9 | 70.6 | 97.7 |
| 8.0 | 61.9 | 93.3 | 99.1 |

BENZENE:NITRIC ACID=1:1 (V/V)

TABLE 4

EFFECT OF BENZENE - TO - NITRIC ACID RATIO

| BENZENE: NITRIC ACID (VOLUME) | PHOSPHORUS RECOVERED (%) | |
|----------------------------------|--------------------------|---------|
| | 1 HOUR | 2 HOURS |
| 2 | 90.2 | 99.5 |
| 4 | 85.1 | 99.6 |
| 6 | 70.8 | 98.5 |
| 10 | 50.1 | 92.9 |

NITRIC ACID=5.3 M

TABLE 5

RECOVERY OF WHITE PHOSPHORUS ADDED TO RIVER WATER

| SAMPLE | PHOSPHORUS CONCENTRATION ($\mu\text{g/l}$) | | |
|--------|--|------------------------|---------------|
| | THEORETICAL | FOUND * | DEVIATION (%) |
| 1 | 0.0011 | 0.0018 | + 63.6 |
| 2 | 0.0022 | 0.0025 | + 13.6 |
| 3 | 0.0044 | 0.0033 | - 25.0 |
| 4 | 0.0110 | 0.0100 | - 9.1 |
| 5 | 0.0440 | 0.0460 | + 4.5 |
| 6 | 0.440 | 0.410 | - 6.8 |
| 7 | 1.10 | 1.05 | - 4.5 |
| BLANK | 0 | 0.0032 \pm 0.0008 | |

* CORRECTION FOR BLANK

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